IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of	:	Dominic A. Cataldo, *et al.*
Serial No.	:	10/816,095
Filed:	:	April 1, 2004
For:	:	Use of Colloidal Clays for Sustained Release of Active Ingredients
TC/AU	:	1615
Examiner	:	Neil S. Levy
Attorney Docket No.	:	BGT 2-007

HONORABLE COMMISSIONER FOR PATENTS
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DECLARATION ON RULE 131

Sir:

Declarant, Dominic A. Cataldo, does declare and state that:

1. His professional credentials and work history are as set forth in his prior declarations, which are of record in the above-identified application;
2. He has read the outstanding Office action mailed on June 11, 2007 and the Ton-That published application cited therein;
3. Ton-That states at paragraph 0048 that he includes hydrophilic polymers and systems in his product, such being aqueous-based systems.
4. Ton-That states at paragraph 0087 that, "Since clay intercalation is usually performed in water, the emulsion and suspension polymerization is natural..."
5. In his opinion, Ton-That does not teach the artisan to conduct clay intercalation in the absence of water, but just the opposite.
6. Further, that the above-identified application was conceived and reduced to practice in this country before November 22, 2002, as evidenced by the copies of his research reports, attached hereto with dates redacted.
7. Specifically, in testing various particles for absorbence of a pesticide for placing in a polymer for forming a barrier to pests, he tested ammonium ion intercalated clays supplied from Nanocor, Inc., as also reported in the data in the Examples in the above-identified application, for example, at p. 14, *to wit*, Nanomer 1.34TCN, 1.30E, and PGV. These ammonium ion intercalated clays were exposed to pesticides (permethrin,

cypermethrin, or fenvalerate), added to a polyester, and evaluated for pesticide release rates. Such clays were determined to last up to 4 times longer than when using carbon black particles.

8. All statements made herein of our own knowledge are true and all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

FURTHER DECLARANT SAYETH NAUGHT.

D A Cataldo

Date: September 5, 2007

Dominic A. Cataldo

[redacted]

Preliminary report on feasibility of the Wellman spun fiber for Reemay replacement.

As we discussed in the past, all of these long-term releasing systems rely on a balance of reservoir size and release rate. Now, if you look at the preliminary data, you can see that we have accomplished one feat in reducing release rate by a factor of four. However, the second driver, reservoir size, is severely hampered in small fibers. The only possible salvation would be if Wellman can spin fibers of 2 mm or greater.

Provide your thoughts and lets talk.

Spun Fiber Simulation Study

Thin 1/8 inch sheets were prepared using 2 polymers, and three TFN loading configurations.
Base on surface area, TFN loading (8%), and anticipated fiber diameters, longevity estimates calculated.
Special nanometer clays were used to increase reservoir holding.

FORMULATI	RELEASE RATE (µg/cm2/day)	PROJECTED SIZE (fiber diameter, mm)	SURFACE AREA (cm2)	ESTIMATED LONGEVIT (years)
PE - w/o CB	182			
PE - w/CB	3.8			
PP - w/o CB	215			
PP - w/ CB	4.2	2	63	2.6
		0.5	25	0.4
		0.1	3.14	0.13
PP- w/clay	1.1	2	63	9.9
		0.5	25	1.6
		0.1	3.14	0.5

SA - $2\pi rh$, set 1 meter fiber length

SA is 63 cm2, vol on 1 m fiber at 2, 0.5 and 0.1 mm are 3.14, 0.20, 0.008 gm (assum density 1)
Loading based on 8% TFN is 251,000, 16,000, and 640 µg

Date: [redacted]

Study No: Production of test pellets for fire ant repellents and insecticides

All products to be based on Solithane S113 100 pbw
Solithane C113 20 pbw
TIPA 15 pbw

Actives at 10% or 13.5 pbv

Warm S113 at 150°F, C113 and TIPA at or near melt temp of 150° F. add active to S113

Pre-blend C113, TIPA, and Active, then add to S113, Mix both components thoroughly

Actives to be used include:	Mp	Vp	WS
Pesticides:			
1) Permethrin - 1		0.07 mPa	
2) Cyfluthrin (hold)		1.6E-5 mPa	
3) Deltamethrin - 4		1.24E-5 mPa	
4) Cypermethrin - 2		2.3E-4 mPa	
5) Fenvalerate - 3			
numbers indicate order of importance			
Attractants:			
1) 2,4-heptadienal			
Repellents:			
1) dimethyl succinate DBE3			
2) diethyl adipate			
3) 1-decanol, decyl alc			
4) 2-nonanol	bp, 100-102		
5) 2-methyl hexanoic acid			

[redacted]

Controls tacky after 4 days – likely poor mixing

[redacted]

Sample preparation for FA attractants and repellents.

Preblended controls as above. Used pipets w and w/o mold release. No hardening after 2 days; probably poor mixing.

Mix as for [redacted] with better mixing. Used S113/C113/TIPA at 100/20/10. Used active at 10 pbv for dimethyl succinate-DBE3, diethyl adipate, 1-decanol, decyl alc, and 2-nonanol; and at 5 pbv for 2-methyl hexanoic acid and 2,4-heptadienal.

Fill 7 10 ml pipets (plastic) to top using vacuum (10 in Hg), mark sample code, stick tip into non-hardening clay fro cure. Pipets pre-sprayed with mold release. Balance of prep poured into weigh boats as test plates.

Old mold release not effective

[redacted]

Prepared pellets from [redacted] evaluated. Working time for all about 30 min. Set,non-tack time below:

A	dimethyl succinate-DBE4,	24 hrs
B	diethyl adipate,	3 da
C	1-decanol, decyl alc,	4 da
D	2-nonanol;	3 da
E	2-methyl hexanoic acid	24 hr
F	2,4-heptadienal.	24 hr

[redacted]

Incorporate FA insecticides into Solithane system, using Permethrin, Deltamethrin, Cypermethrin and Fenvalerate. (no Delta – insufficient)

Used [redacted] procedure with 10 pbv active (14 mL). Filled 2 each 10 mL plastic pippets, and 3 plates. Used new mold release, #205 from BP.

		Set time	Size
H	Permethrin		
I	Cypermethrin		
J	Fenvalerate		
K	MeTilgate (half batch)	1 mL active	
L	BuHex (half batch)		1 mL active

Set time was 48 hrs

[redacted]

Shipped Samples to ARS for evaluation.

Shipped samples to Gulfport for evaluation

Designations: 24 each of cock roach; 2x24 each of FA

FA Repellents
- 02-74-A Dimethyl succinate
- 02-74-B Diethyl adipate
- 02-74-C 1-decanol
- 02-74-D 2-nonanol
- 02-74-E 2-methyl hexanoic acid

FA Attractant
- 02-74-F 1,4-heptadienal

FA Pestcides
- 02-74-H Permethrin
- 02-74-I Cypermethrin
- 02-74-J Fenvalerate

Cockroach Attractants
- 02-75-K Me tilgate

[redacted]

02-75-L Butyl hexanoate

[redacted]

Reviewed data from ARS. Duration of devices short, indications of polymerization of actives with polymer.

Next set to contain actives within clays. Clay dried for 72 hrs at 120°C. Vials prepared containing x gm clay Attap, active added until wetted, after first wet pulled vacuum to clear clay air spaces.

Active*		Tare	Clay wt	Act wt	Act/gm	Total gm
02-76-M Dimethyl succinate cloudy	14.97	19.99	27.66	0.6	12.69 (5)	
02-76-N Diethyl adipate	15.11	20.11	27.53	0.58	12.43 (5)	
02-76-P 1-decanol	15.04	19.90	25.36	0.53	10.32 (5)	
02-76-Q 2-methyl hexanoic acid		15.14	19.82	24.83	0.52	9.69 (4)
02-76-R 1,4-heptadienal	15.06	19.51	23.77	0.49	8.71(4)	
02-76-S Permethrin	15.04	19.85	25.78	0.55	10.74 (4)	
02-76-T Cypermethrin gummy	15.07	19.63	26.90	0.61	11.83 (5)	
02-76-U Fenvalerate gummy	15.16	20.15	27.29	0.59	12.13 (5)	

* all others seem to blend OK

Formulations (# = gm blended)

S113/C113/TIPA at 100/20/10

Mix S113 (20 mL + 4 mL C113), blend active at 4-5 gm as above limits, then add 2 mL TIPA, wait for new catalyst

Polyester

28.3g gm/oz (31.3 gm/ fl oz or 1.1 gm/mL) polymer, mix 4-5 gm active as above, add 15 drops catalyst. Prepared 10 am n 10/27/02, set time ////

Solithane designated 02-76-MA – DID NOT DO, changed linker
Polyester designated 02-76-MB, Prepared Oct 24, 2002, Bottled as below.

Solithane

02-76-MA	Dimethyl succinate
02-76-NA	Diethyl adipate
02-76-PA	1-decanol
02-76-QA	2-methyl hexanoic acid
02-76-RA	1,4-heptadienal
02-76-SA	Permethrin
02-76-TA	Cypermethrin
02-76-UA	Fenvalerate

Polyester Produced [redacted] used red dye

02-76-MB	Dimethyl succinate (5)
02-76-NB	Diethyl adipate (5)
02-76-PB	1-decanol (5)

[redacted]

02-76-QB	2-methyl hexanoic acid (4)
02-76-RB	1,4-heptadienal-A (4), dissolved Polyester tray
02-76-SB	Permethrin (4)
02-76-TB	Cypermethrin (5)
02-76-UB	Fenvalerate (5)

[redacted]

Prepared alternate absorbants

Nylon-6 (nanocor)
Letter designations for actives from above, soak

1) 02-77-SM, 5gm N6		20.03 w/cap
2) 02-77-SN, 5 gm N6	20.01	
3) 02-77-SP, 5 gm		20.04
4) 02-77-SV, 5 gm		20.04

V= Nonanol

Duplicated with pellethane (7-10) and santoprene ([redacted]), on [redacted]

** Pellethane absorbed N>M>P=V
Nylon 6 and santoprene little absorp

[redacted]

Dried (110°C/24 hrs) Montmorillinite clays from Nanacor on [redacted]

N1.34TCN		Active	mL Act added
6) 2.34 gm,	tare 17.48 w/cap	M	4, good swell*
7) 2.49	17.54	P	4, slightly too much
8) 2.30	17.36	S	3, good*
N1.30E			
9) 2.43	17.48	M	4, good*
10) 2.28	17.35	P	4, good*
11) 2.67	17.93	S	4, good*
15) V , Nonanol w/N1.30	16.90	V	3, good*
NPGV			
12) 3.04	17.98	M	4, no swell, too much
13) 4.34	19.37	P	3, swelled, too much*
14) 3.88	18.94	S	2, too much

Cut and packaged samples of Polyester

02-76-MB	Dimethyl succinate (5)
02-76-NB	Diethyl adipate (5)
02-76-RB	1,4-heptadienal-A (4)

Mixed 25 mL of S113 with 5 mL of Cog 115 or 253. Did not set.

[redacted]

[redacted]

Preparation of new formulations

Solithane/Tipa-C113
Mix S113 (20 mL), blend active at 4-5 gm as above limits, then
add 2 mL TIPA + 4 mL C113.
W/ pest from 02-76, N, P
Hold study

Solithane/ Cogna (U)

Blend preheated S113 with active, then Cog 115 or 253. Use 15 gm S113 and 40 gm of 115 or 253. Blend active to S113, first.
W/ 6 clays with above *
W/ [redacted] clays

Tried 1gm S113 with 2.65 gm 115 and 253. Only 115 setup. USE

From [redacted] actives +clay

02-77-MDimethyl succinate (5)
02-77-N Diethyl adipate (5)
02-77-P 1-decanol (5)
02-77-Q 2-methyl hexanoic acid (4)
02-77-R 1,4-heptadienal-A (4)

02-77-S Permethrin (4)
02-77-T Cypermethrin (5)
02-77-U Fenvalerate (5)

No polymerization ******* need more 115????

02-78- M6
02-78-S8
02-78-M9
02-78-P10
02-78-S11
02-78-P13
02-78-S14
02-78-V15 Nonanol Alc

[redacted]

[redacted]

Redo with M, N, P, V, and S using N1.30E, at rate of 1 clay - 2 parts active.
Actual was 8 gm caly to 18 ml of active. Only M slightly liquidy, all others thick.

Use Grey epoxy, solvent urethane, and Silicone rubber (dent). Prepare 3 reps of each active and polymer. Mix ratio at convenience.

[redacted]

U and D manufactured 11/16, in air till [redacted]
Compounds M and N reacted with polystyrene boats
Sil 102 manufactured on [redacted], in air till all no set, exc Permethrin/tacky
Epoxy (Devcon) manufactured on [redacted]
Actives (A) include clay and active